

CRITERIA FOR COMPOSITION AND
PROPERTIES OF HOT-APPLIED ASPHALT PIPELINE COATINGS

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Asphalt has been used to coat buried pipelines for many years.

It was included in the API burial tests which were started in 1922 [1]. The asphalt coatings of that era were, for the most part, modifications of other products offered for pipe coating. During the last 20 years efforts have been directed toward producing asphalt base pipe coatings with the functional properties required of a coating in this specialized service.

To place this paper in proper context, it should be pointed out that corrosion mitigation on a buried pipeline nearly always involves a protection system. This protection system can consist of both a coating system and cathodic protection. The coating system can contain as many as four components, i.e. primer, hot-applied coating, embedded wrappers and outer wrappers. The first two coating components are the subject of this paper and the total protection system will be considered only as it affects the performance of these two asphaltic components.

The purpose of this paper is to review criteria for establishing composition and properties of asphaltic primers and coatings. New developments in prevention of disbonding through use of chemical additives will be presented. The total performance of a pipe coating system involves three separate use periods: application to the pipe, handling and storage of the pipe, and in-ground service of the pipe. Each of these use periods imposes functional requirements on the coating and exposes it to potentially deteriorating environments or forces. Both the functional requirement and resistance to deterioration must be considered in establishing criteria for coating properties.

Two important coating performance areas are retention of adhesion in the presence of cathodic protection and resistance to deformation by mechanical and soil stresses. These are the two performance areas which are treated in the most detail.

PROPERTIES OF HOT-APPLIED ASPHALT PIPE COATINGS

A long list of physical properties is used to describe hot-applied asphalt coatings. Some of the areas they describe are: 1] uniformity; 2] coverage rate; 3] application characteristics; 4] coating performance during pipe storage and handling; and 5] coating performance after the pipe is in service. Most of the properties now used to describe hot-applied asphalt coatings together with typical values are shown in Table I. Many of the test procedures are described by ASTM or some other specification writing agency. Those which are not so described appear in the appendix.

Broadly described, hot-applied asphalt coatings consist of an air-blown asphalt [75-85%] and a finely divided mineral filler [15-25%]. The mineral filler is used mainly to impart toughness. Most coatings on the market today fall within a 225 to 275°F softening point range and are quite hard [4-10 penetration at 77°F] at ambient temperatures.

The main function of the primer is to produce a strong adhesive bond. Therefore, they are formulated to have the low viscosities [70 centistokes at 77°F is typical] required for good wetting properties. Primers vary quite widely in composition. Solvent contents in the range of 55% by weight are needed to obtain the low viscosities. The film forming portion is usually mostly asphalt. Typical properties are shown in Table II.

Application Properties

Important application characteristics of a hot-applied pipe coating are:

1. Degree of change in composition and properties caused by heating and handling during application.
2. Fire hazard and fumes associated with its application.
3. Set time after application.
4. Its behavior during electrical inspection.

The thickness of coating applied to the pipe is largely dependent on the viscosity of the coating. Because of this, the coating's temperature-viscosity curve becomes an important consideration. In order to obtain thickness of $3/32$ " on the pipe, viscosities in the range of 100-200 centipoise are usually required. The temperature required to attain this viscosity varies from coating to coating. Thus, a coating's volatility, thermal stability, settlement ratio, and flash point should be considered in light of its temperature-viscosity curve.

Filler settlement ratio is a function of the viscosity of the fluid, density of the particle, diameter and shape of the particle. Since viscosity is set by the film thickness desired, the characteristics of the filler particle must be controlled to hold settlement to the desired level. The amount flash point exceeds application temperature is indicative of both fire hazard and fuming that can be expected. For practical reasons, electrical inspection voltages in the range of 25,000 are sometimes used. For a 50 mil. coating this is 500 volts per mil. of thickness. In order

to have a 2 to 1 safety factor, a coating should resist electrical breakdown up to 1000 volts per mil.

Handling and Storage Properties

Once a good coating application job has been achieved, what happens during the storage and handling period of the coated pipe determines the effectiveness of the coating when it is placed in service. The coating on stored pipe may experience temperatures as low as 0°F during the winter in northern climates and as high as 160°F when exposed to the sun during the summer in southern climates. A 100°F drop in surface temperature within 24 hours can be experienced in a given location. A coating must be able to relieve the shrinkage stresses developed during this cooling, otherwise it will crack. The low temperature crack test is used as an indicator of performance in this area. The impact test is used as a measure of mechanical abuse coated pipe can withstand in handling without film fracture. The sag test is used as an indicator of resistance to flow when exposed to summer sun in southern climates.

In-Service Performance Properties

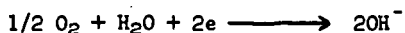
Now let us consider the properties that are indicative of a pipe coating's performance in service. Some of these are: 1] cathodic disbonding resistance; 2] flow resistance; 3] low temperature crack resistance; 4] electrical resistance; and 5] water absorption. The relative importance of each of these varies widely from one installation to another. For example, cathodic disbonding resistance of a coating is of little

importance unless the pipe is cathodically protected. The importance of flow resistance is related to the nature of the soil surrounding the pipe and to the pipeline operating temperatures.

Cathodic Protection

Since cathodic protection is widely used to complement coatings on buried structures, it is important to consider their efficiency as used together and the effect of one upon the other. Cathodic protection involves impressing a negative potential on the structure to be protected. The protected structure thus becomes the cathode of a large electrochemical cell. Some of the chemical reactions that are promoted by its negative character are:

In aerated nearly neutral soils oxygen reduction can occur:



In acid soils the hydrogen ion is reduced:



Because the hydrogen ion has such a great affinity for H_2O [2]

the more complete picture is:



This is the primary reaction occurring in neutral and alkaline soils also. The H^+ ion becomes available from ionization of H_2O . Since it has a more noble position in the electromotive series than other cations usually present, it is preferentially reduced. Depletion of H^+ concentration at the cathode surface produces additional H_2O ionization. The over-all result is the formation of H_2 gas at the cathode and an ever increasing OH^- concentration in its vicinity. Some of the metal cations precipitate as hydroxides. Others react further such as with CO_2 to precipitate as carbonates.

The effect of cathodic protection on loss of coating adhesion has been considered by others [2,3,4]. Since the use of cathodic protection in conjunction with a coating is based on the premise that it should protect the metal at coating imperfections, our laboratory investigations employ specimens with intentional "holidays" [holes in the coating]. The two procedures used are described in the Appendix. The short-term test is usually run seven days and is used as a screening test. The long-term test is run from 30 to 270 days.

Figure 1 is illustrative of a good and poor performing coating system in the short-term test. Usually there is loss of coating bond with no apparent displacement of the coating by hydrogen. Occasionally slight displacement is noticed. On one occasion a coating system disbonded completely and hydrogen pressure lifted it approximately 1/4" from the panel surface at the center. The extension was by cold flow without rupture of the coating. This is illustrated in Figure 2. Pictures illustrating a good and poor performing system in a 270-day cathodic disbonding test are shown in Figure 3. [All photographs were taken after disbonded coating was mechanically removed.]

Current drain to the test specimen is rather irregular and often does not correlate with the amount of disbonding. A graph illustrating the order of magnitude of current drain for three coatings that showed different degrees of disbonding is shown in Figure 4.

The use of additives in the primer for improving the cathodic disbonding resistance of a poor performing primer-coating system was investigated. The additives were used at a concentration of 1% by weight. Additives

which improved resistance to cathodic disbonding included an imidazolinium chloride [7,8], an amine [9], and a substituted pyridine [10]. Disbonding resistance was reduced by three diamines and two commercial anti-strip additives [adhesion improvers for road asphalts]. A listing of materials tested and results obtained are shown in Table III.

Our work shows that cathodic disbonding is affected both by the primer and the coating used over it. However, as would be expected the primer is the most important component in controlling this property. Data to illustrate this is shown in Table IV. Cathodic disbonding can be cut in half by going from a poor to good performing coating and can be cut ten fold by going from a poor to good performing primer.

Flow Properties

A buried pipe coating is subjected to the pressures caused by the weight of pipe and its contents and to pressures generated by expanding and contracting soil. If it flows under these pressures, the pipe is no longer uniformly coated. A blunt rod deformation test is widely used to rate flow resistance of pipe coatings. This test consists of measuring the deformation [in 0.001 inches] at given time periods caused by the weight of a stainless steel rod 1/4" in diameter and of such length as to produce a pressure of 2 psi. A plot of values obtained in this test vs. penetration [both at 150°F] is shown in Figure 5. There is very little correlation. Both measure flow resistance but at greatly different rates of shear. For this reason these two tests would give proportional values only for those bituminous compositions of equivalent shear susceptibility.

Since loading is lower [lower shear rate] in the rod deformation test, it will give lower values with increasing shear susceptibility for materials of equivalent penetration at the temperature under consideration.

Water Absorption and Electrical Properties

Water absorption and electrical properties [5] of asphalt is discussed in detail by the authors in a recently issued book edited by Hoiberg. Some important points related to this subject are:

1. Pure asphalt absorbs water at a very slow rate
2. The prime consideration is whether or not the physical nature of a pipe coating is materially changed by the amount of absorption which occurs.
3. Electrical resistance is reduced by water absorption but even for high absorbers current drain through the coating would be quite small on a cathodically protected pipeline [6].

SUMMARY

In assessing the performance of a pipeline coating, three time periods during its utilization must be considered. These are: 1] its application to the pipe; 2] the pipe storage and handling period; and 3] the pipeline in service period. The latter period is the one of major importance but a coating's usefulness during this period is greatly affected by its behavior in the first two. Cathodic protection is widely used as a complementary means of corrosion mitigation on buried structures and its disbonding effect on coatings is an important consideration.

Cathodic disbonding is affected by composition changes in both the primer and the hot-applied coating. It is most sensitive, however, to changes in the primer. The flow properties of hot-applied asphalt coatings are an important factor in their performance. It is possible to have materials varying widely in shear susceptibility, therefore, meaningful laboratory tests must employ loading rates of the same magnitude and at the same temperature that will be encountered in service.

REFERENCES

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8. U.S. Patent No. 3,070,455.
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10. U.S. Patent No. 3,070,523.

APPENDIX

TEST PROCEDURES FOR HOT-APPLIED ASPHALT PIPE COATINGS

CATHODIC DISBONDING TESTS

I. Preparation of Electrolyte for Cathodic Disbonding Tests

The synthetic sea water used as the electrolyte in the disbonding test conforms to military specification MIL-L-21260 and has the following composition:

<u>Ingredients</u>	<u>g./l. of H₂O</u>
Magnesium Chloride - 6H ₂ O	11.0
Anhydrous Calcium Chloride	1.2
Anhydrous Sodium Sulfate	4.0
Sodium Chloride	25.0

The pH of the solution shall be adjusted to a value of 8.0 to 8.2 by addition of a 5% solution of sodium carbonate or a 5% solution of hydrochloric acid, whichever is necessary. All chemicals used should conform to ACS standards for analytical reagents chemicals.

II. Preparation of Test Specimens for Short-Term Cathodic Disbonding Test

A. Introduction: The following is the standard procedure for preparation of specimens for short term cathodic disbonding tests. Specimens are prepared from 2-3/4" x 5-7/8" x 0.035" open hearth steel panels.

B. Preparation - Procedure:

1. First day - Metal surface preparation and application of prime coat.

- a. Wipe panels free of oil.
- b. Slush panels in nearly boiling 300/360 b.r. naphtha.
- c. Sandblast both sides of panel. For blasting, a sharp, dry sand having a minimum of 50% retained on a No. 60 sieve shall be used with an air pressure of not less than 50 pounds per square inch.
- d. Solder copper lead wire to panel using rosin core solder.
- e. Brush free sand from panel with stiff brush.
- f. Apply primer to panel using clean brush for each primer.
- g. Allow panels to dry overnight.

At all times during the above procedure precautions should be taken to prevent contamination of test side of panel. The test side of the panel is designated as the side opposite the soldered wire. The primed panel should not be allowed to stand for more than 24 hours before application of hot enamel.

2. Second day - Application of hot enamel.

- a. Apply hot enamel to test side of panel to a thickness of 55/65 mils, using a plaqueing machine.
- b. Incline panels on 3 oz. ointment cans with coated side down and flow coat back side of panel with hot enamel.

- c. Dip all edges of coated test specimens in hot enamel to prevent accidental damage at weak points. Cool panels in ice water immediately after dipping to prevent "running" of the enamel.
- d. Drill a 1/16" "holiday" through the coating on the test side of the prepared test specimen.

Care must be exercised in coating the test side of the primed panel with hot enamel. The coating must be in the 55/65 mil range, otherwise reproducibility of the test will be affected.

III. Preparation of Test Specimens for Long-Term Cathodic Disbonding Test

- A. Introduction: The following is the standard procedure for preparation of specimens for long-term cathodic disbonding tests. Specimens are prepared from 2' lengths of 2" I.D. steel pipe threaded on each end to receive a plastic coupling on one end and a plastic cap on the other.

- B. Preparation - Procedure:

1. First day - Metal surface preparation and application of prime coat.
 - a. Solder copper lead wire to inside of pipe.
 - b. Sandblast pipe surface using a sharp, dry sand having a minimum of 50% retained on a No. 60 sieve shall be used with an air pressure of not less than 50 pounds per square inch.
 - c. Brush free sand from sandblasted pipe using stiff brush.
 - d. Apply primer to pipe using clean brush for each primer.
 - e. Allow pipes to dry overnight.

At all times during the above procedure precautions should be taken to prevent contamination of freshly sandblasted pipe surface. The primed pipe should not be allowed to stand for more than 24 hours before application of hot enamel.

2. Second day - Application of hot enamel.
 - a. Screw the coupling and cap to their respective ends of the pipe.
 - b. While pipe is mounted horizontally and rotating at a constant speed, flow coat with hot enamel to a thickness of 90 + 5 mils. Bring hot enamel up over plastic coupling and cap on each end.

- c. After coating has cooled, dip capped end of pipe into molten enamel to effect a water-tight seal.
- d. Drill four 1/16" dia. holidays in the coating equidistant apart and offset 90° from each other along the pipe length.

IV. Operation of Test Apparatus

- A. Test Apparatus: The attached Figure I describes a single cathodic disbonding test circuit and the equipment used for measuring potential and current drain. Sixty 100 ohm rheostats and insertion jacks are mounted on a central panel board and connected in parallel to a single anode. Use of a phone plug enables taking current and voltage readings without interrupting current drain to the test specimen. The constant voltage transformer may be dispensed with if an unvarying source of a.c. is available.
- B. Test Procedure: The test specimens prepared as described above are immersed in the solution of synthetic sea water around an anode and subjected to a potential of -1.50 volts with reference to a saturated calomel electrode. Current readings are made and recorded, and the voltage is adjusted to -1.50 volts weekly on each specimen. Any addition of water to compensate for evaporative losses and necessary pH adjustments are also made at this time. The equipment used in the cathodic disbonding test is illustrated in Figure 1.

At the end of the prescribed test period the specimen is removed from the bath and examined for disbonding beyond the intentional damage. The loosened coating is removed to the point where firm bond still exists. The disbonded area is then measured and recorded in square inches.

High Temperature Sag Test

Preparation of test plates: Test plates shall be mild steel of 12" x 4" x 3/16" size. One side of each plate shall be freshly sand-blasted to a uniform steel gray surface, completely removing stains, rust and mill scale.

For blasting, a sharp, dry sand having a minimum of 50% retained on a No. 60 sieve shall be used with an air pressure of not less than 50 pounds per square inch.

Application of the coating: The plates shall be primed with the recommended primer for the coating under test. After the primer has cured as recommended by the manufacturer. The molten hot coating shall be applied by pouring on the plates supported at an incline of 10 degrees. Temperature of the coating shall be adjusted to obtain a film thickness of $3/32" \pm 1/64"$.

Test procedure: Reference lines drawn with wax pencil or chalk shall be drawn at three-inch intervals parallel to the 4" side. The coated plates shall then be placed vertically in a test oven with the scribed lines horizontal at the desired test temperature for the desired length of time. The oven shall be capable of controlling within $\pm 5^{\circ}\text{F}$ of the test temperature.

After completion of the test the plates shall be removed from the oven and allowed to cool to room temperature. The lowest point on each of the lines shall be checked against the reference point on the edge of the plate and the difference reported as sag distance.

Low Temperature Crack Test

Preparation of test plates: Same as for High Temperature Sag Test.

Application of Coating: Same as for High Temperature Sag Test.

Test Procedure: Place the coated plates in an air bath capable of controlling at the desired test temperature $\pm 5^{\circ}\text{F}$. After a period of 6 hours at test temperature, remove the plates and allow them to warm to room temperature and examine for evidence of cracking or disbonding. A jump spark holiday detector may be used to examine for cracking.

Resistivity, Ohm-cm.

Scope: This procedure is intended for determining the volume resistivity of pipe coating materials having values up to 10^{17} ohm-cm. It is intended only for those materials capable of being formed in films and sufficiently hard to be handled at 75°F without damage.

Equipment: Film former - Carver Press equipped with heated platens
240 Regulated High Voltage Power Supply -
Keithley Instruments, Cleveland, Ohio
6105 Resistivity Chamber - Keithley Instruments
Decade Shunt - Model 2008 Keithley Instruments
Ammeter - 0-10 scale - Hooked through decade shunt -
Keithley Instruments.

Procedure: A. Initial resistivity

Films, 50-70 mils thick, are formed by a suitable means, e.g. Carver Press. Hot forming of the films between Teflon Paper will enable easy release. Circular test specimens, 3 inches in diameter, are then cut from the films. Measure and record the specimen thickness in cm.

Place the test specimens between the electrodes in the resistivity chamber and impress a 500-volt potential across the film. Wait 10 minutes to allow the current across the film to equilibrate and note the amperage.

Using Ohm's Law, calculate the film resistance:

$$E = IR$$

Where: E = potential across film, volts
I = current drain across film, amps.
R = resistance of film, ohms

The value for R is then inserted in the volume resistivity equation:

$$P = \frac{RA}{L}$$

Where: P = volume resistivity of film, ohm-cm.
R = film resistance, ohms
A = area of electrode face, cm.²
L = film thickness, cm.

B. Resistivity after immersion:

Immerse the test specimens in distilled water for the desired length of time. After such time, remove the specimens, pat dry with an absorbent tissue, and allow to air dry for one hour. After one hour proceed with the volume resistivity determination outlined above.

Voltage Breakdown

Coating is applied to a steel plate such that coating thickness of 5 to 10 mils is obtained. Voltages greater than 1000 times the film thickness in mils is applied across the coating with an electric holiday detector equipped with a variable voltage regulator.

Water Absorption

Test panels are prepared by pouring the molten enamel down the inclined face, at approximately 30° from the horizontal, of the polished aluminum test panels, each 2-3/4" x 5-7/8" of 20 gauge metal. By balancing the inclination of the panel and the temperature of the coating against the viscosity of the test material, test films of 90 to 100 mils thickness can be obtained on both sides of the panel. After cooling, the edges of the coating are trimmed at an angle of 45° and the edges then coated uniformly by hand dipping in the enamel. The panels are weighed initially and again after 35 weeks' immersion in water, the water absorption being reported as percentage by weight of initial weight of film.

TABLE I

HOT-APPLIED ASPHALT PIPE COATINGS
TYPICAL PROPERTIES AND THEIR SIGNIFICANCE

Property Information is use for:	Property	Typical Value
Indicating Application Characteristics	Flash Point, ASTM D-92	575+
	Temperature-Viscosity curve, Brookfield viscometer	---
	Voltage breakdown resistance, volts per mil ¹	1000+
	Penetration at 150°F, ASTM D-5	30
	Settlement ratio [filler], AASHTO T 109-42 [modified]	1.10 at 400°F
Indicating Coated Pipe Handling and Storage Characteristics	Flow resistance [rod deformation], Asphalt Institute ²	15 mils, 6 hrs., 115°F
	Low temperature crack resistance, Asphalt Institute ³	Pass to 0°F
	High temperature sag resistance, Asphalt Institute ³	160°F+
	Impact resistance, [Southern California Gas Company procedure [modified]]	Pass to 32°F
Indicating Coating Performance in Service	Flow resistance [rod deformation], Asphalt Institute ²	15 mils, 6 hrs., 115°F
	Low temperature crack resistance, Asphalt Institute ³	Pass to 0°F
	Cathodic disbonding resistance, sq. inches ¹	0.03-0.5
	Electrical resistance, ohm-cm ¹ Water absorption, wt. percent ¹	7-day test ⁴ 1 x 10 ⁻⁵ + 1.0 in 35 weeks
Indicating Uniformity	Penetration at 77°F, ASTM D-5	7
	Softening point, ASTM D-36	240
	Ash [mineral filler], wt.%, ASTM D-482 [modified] Weight, pounds per gallon, ASTM D-71	20 10
Determining coverage rate per unit of weight	Weight, pounds per gallon, ASTM D-71	10

¹Test procedure given in the appendix²Asphalt Protective Coatings for pipe lines, C.S. No. 96, 2nd edition, The Asphalt Institute, Dec. 1958³Asphalt Protective Coatings for pipe lines, C.S. No. 96, The Asphalt Institute, May 1954, now superseded. Test procedure given in the appendix.⁴This property is quite dependent upon the primer used.

TABLE II

PRIMER FOR HOT-APPLIED ASPHALT
PIPE COATING - TYPICAL PROPERTIES

<u>Property</u>	<u>Typical Value</u>
Flash Point, T.O.C., °F	95
Viscosity, centistokes at 77°F	70
Solids Content, wt. %	45

TABLE IV

RELATIVE EFFECT OF COATING
AND PRIMER ON CATHODIC DISBONDING

<u>Primer</u>	<u>Coating</u>	<u>Disbonded area</u> <u>7 days, -1.5 volts,</u> <u>sq. in.</u>
I [poor]	A [poor]	0.52
I [poor]	B [good]	0.26
II [good]	A [poor]	0.04
II [good]	B [good]	0.03

TABLE III

EFFECT OF ADDITIVES TO PRIMER ON SEVEN-DAY CATHODIC DISBONDING

[Additive used at 1% by weight, Voltage - 1.5 to sat. cal. electrode]

Trade Name	Additive Compound Type	Disbonding		
		Area, in. ² Primer + Additive	Area, in. ² Primer [Control]	Ratio, Additive Primer/ Control
Nalquat G-8-13	1,[2-Hydroxyethyl]- 2-heptadecenyl, 1-benxyl- 2-imidazolinium chloride	0.332	0.695	0.478
Armeen C	Coco amine	0.313	0.441	0.705
Duomeen S	Soya, 1,3-propylene diamine	1.92	0.609	3.16
Duomeen T	Tallow, 1,3-propylene diamine	1.15	0.490	2.35
Ethoduomeen T12	Alkoxyated alkyl 1,3-propylene diamine	2.78	0.706	3.93
PAP 250	Polyalkyl pyridine	0.397	0.554	0.715
Nalclad RL-1	Commercial anti-strip additive	0.92	0.410	2.24
Pave	Commercial anti-strip additive	1.42	0.465	3.05

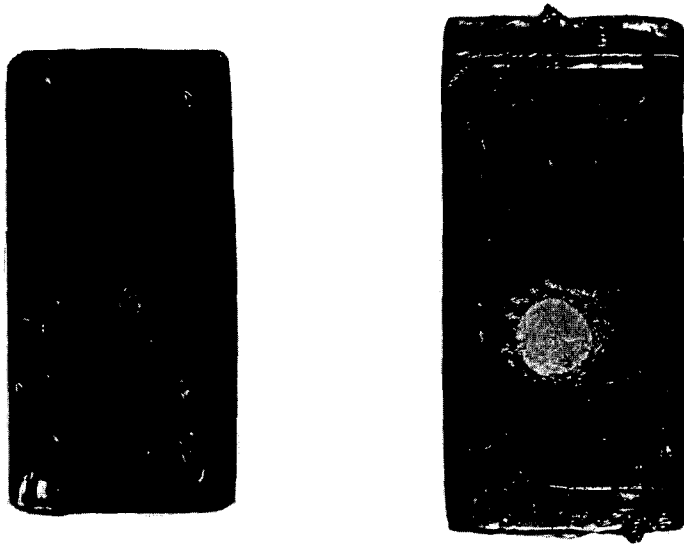


FIGURE 1

GOOD AND POOR PERFORMING COATING SYSTEMS
IN A 7-DAY CATHODIC DISBONDING TEST

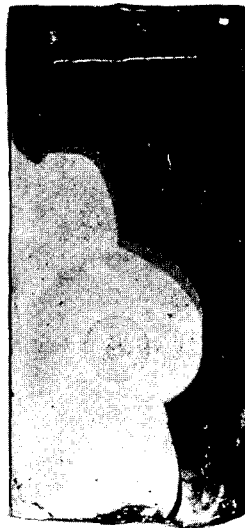
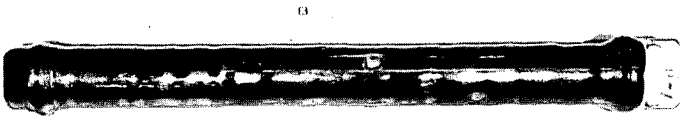


FIGURE 2

COATING COLD FLOW FROM HYDROGEN PRESSURE
IN CATHODIC DISBONDING TEST

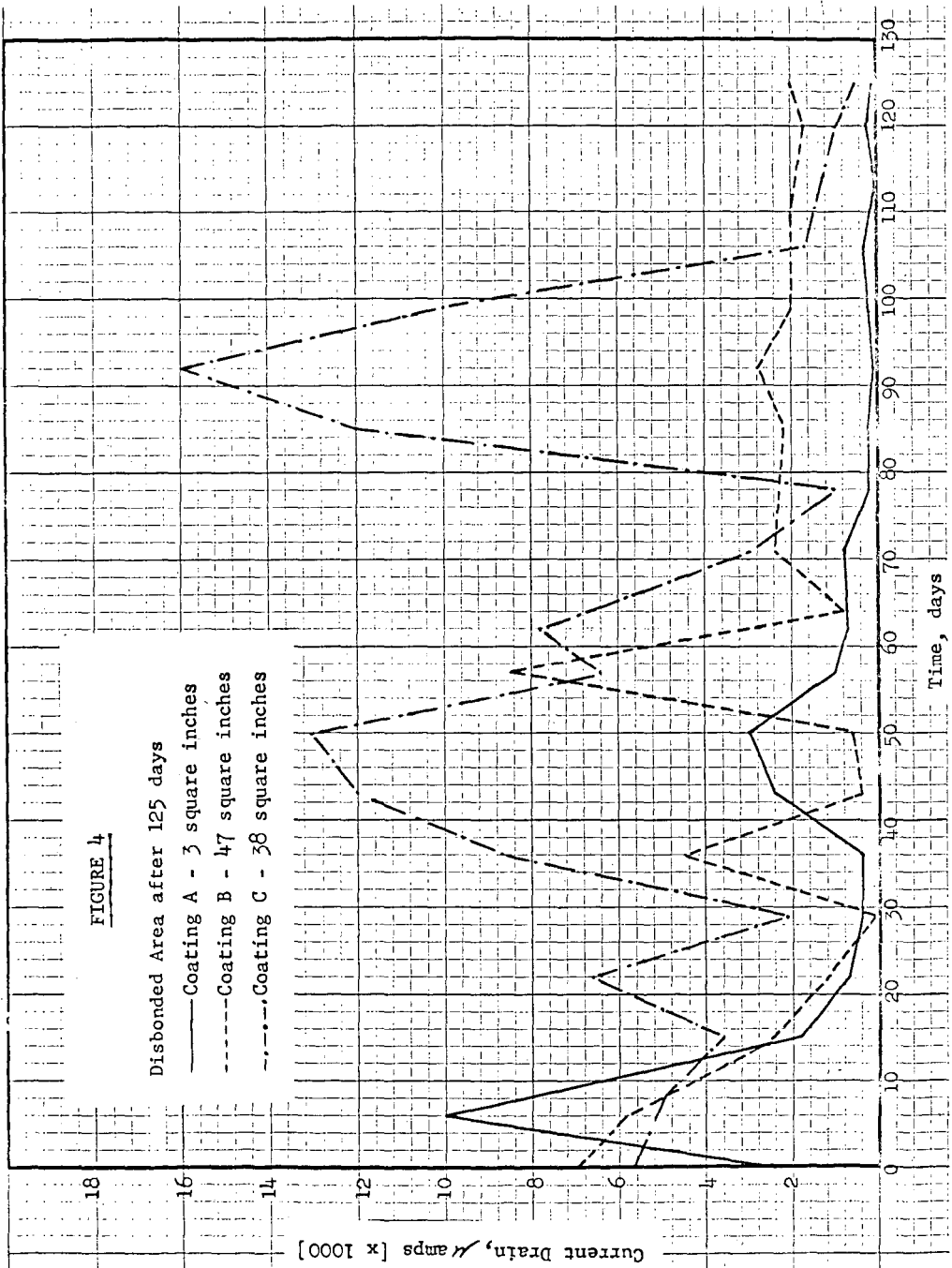
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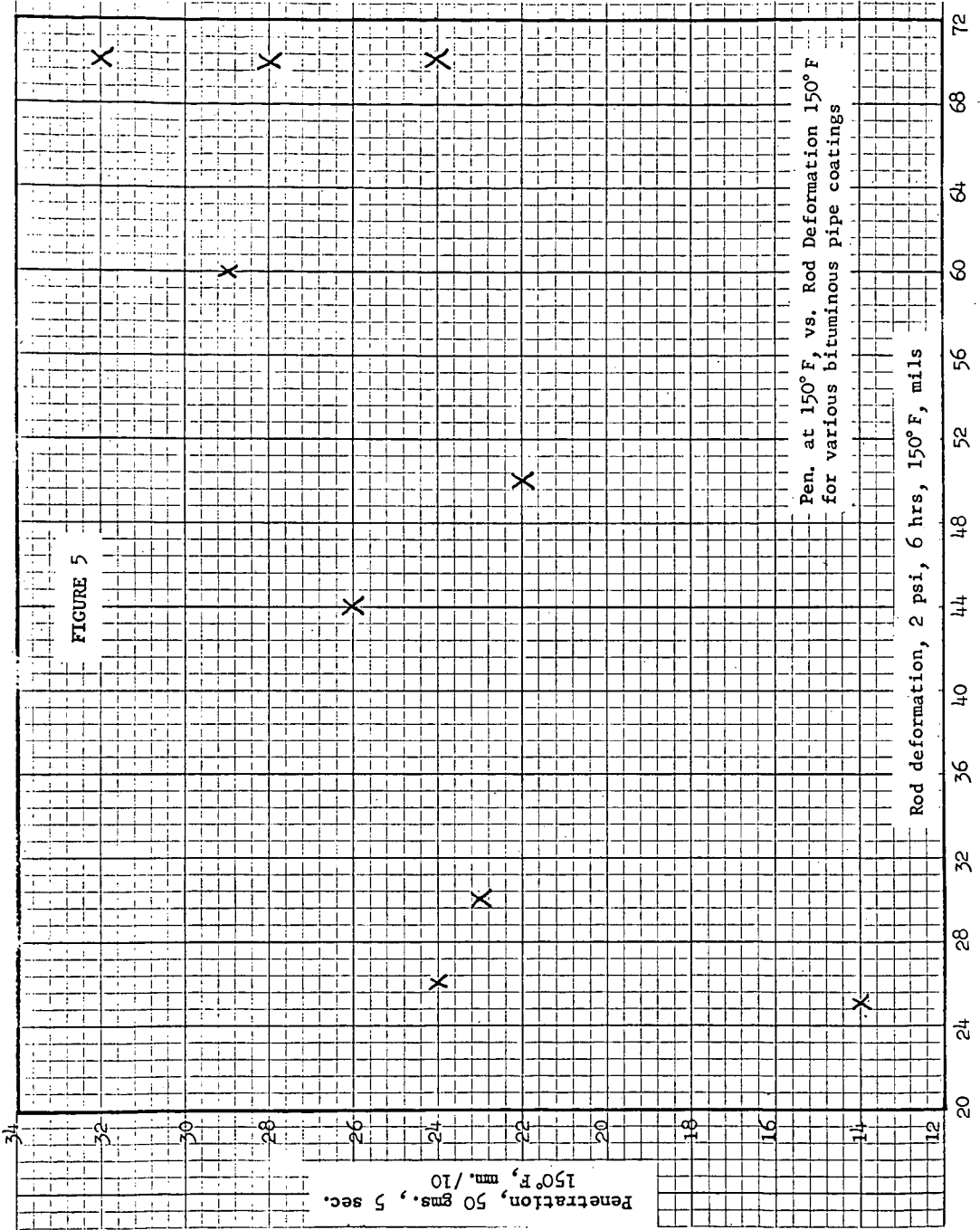


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FIGURE 3

GOOD AND POOR PERFORMING COATING SYSTEMS
IN A 270-DAY CATHODIC DISBONDING TEST





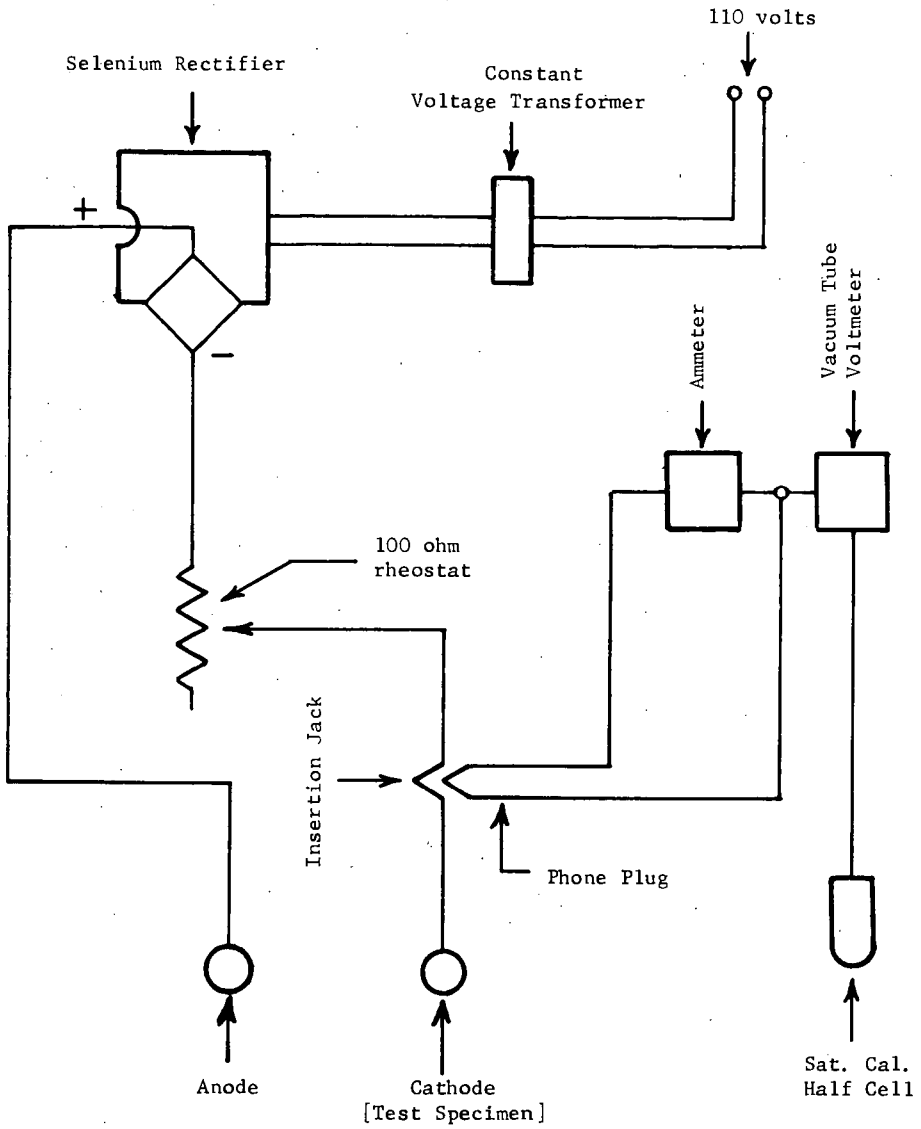


FIGURE I - APPENDIX